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## **Photo-ignition of Carbon Nanotube for Ignition of Liquid Fuel Spray and Solid Fuel**

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### **Abstract**

We have studied the ignition of liquid fuel and certain solid fuels by the photoignition of single wall carbon nanotubes (SWCNTs). Our investigation includes the effect of solid additives such as aluminum nano-particles and solid oxidizers such as ammonium perchlorate (AP) on the photoignition characteristics. We have shown that by mixing CNT with other nanoparticles and powdered material, the ignition parameters such as; burn temperature and burn duration can be tailored to meet ignition requirements. We believe this approach in photoignition of a fuel spray and a solid fuel provides a suitable method for ignition of liquid rocket engines and solid rocket motors. Among the advantages of this approach are a compact, low weight, and robust ignition method and it enables volumetric distributed ignition of fuel sprays.

### **Introduction**

The first report on photoignition of single wall carbon nanotubes single wall carbon nanotubes SWCNTs by a camera flash appeared in 2002.<sup>1</sup> It was suggested that Fe nanoparticles within SWCNTs play an important role in the photoignition process.<sup>2</sup> Subsequently, others reproduced some of the aforementioned results and offered that SWCNTs could be used as ignition agents for a variety of liquid fuels, including those of interest in liquid rocket engines.<sup>3-5</sup> It was also proposed that SWCNTs could facilitate distributed ignition of liquid sprays. In an earlier work, we showed that liquid fuel can be ignited through photoignition of SWCNTs.<sup>6</sup> Other applications of photoignition of SWCNT has also been reported.<sup>7-9</sup> More recently other reports on the photoignition of the gaseous fuel and air mixture showed distributed ignition.<sup>10,11</sup> There has been a recent report on the photoignition of graphene oxide for fuel ignition applications.<sup>12</sup> In this report, we will describe the experimental procedure the results for achieving photoignition of a liquid spray and a solid fuel by SWCNTs.

### **Experimental Setup and Instrumentation**

Samples of the carbon nanotubes (CNTs) with different nanoparticle Fe content that was used in this study were from Unidym Inc., Houston, Texas. The samples were as grown ("raw") CNT and contained different impurities (mostly Fe and carbon particles) and the CNT content is be predominantly SWCNT. The term CNT is used for the samples in this report in order to indicate that they were not purified SWCNT samples. Except for the photoignition, most other applications require high concentration of SWCNT and low concentration of Fe, which explains the need for the purification process. Purified samples show a considerably higher photoignition threshold.

Two experimental setups were utilized for the reported results, one for quantitative measurements of photoignition criteria for CNT and the second one for imaging photoignition dynamics. The first experimental setup, referred to as CNT ignition setup, consists of a Canon camera flash, model Speedlight 580 EXII compact Xe-arc, as ignition light source, a pulse energy meter from GenTec, SUN series EM-1 with ED-500 detector head. The light source provided a 22-step intensity setting with 0.1 to 7-ms duration at the lowest to the highest light setting with a maximum of about 5 J/cm<sup>2</sup> per flash.

The light is coupled from the source to the sample through a 3'x1/2" quartz fiber optic light guide from Sunoptics Technologies along with an XYZ linear translation stage for continuous

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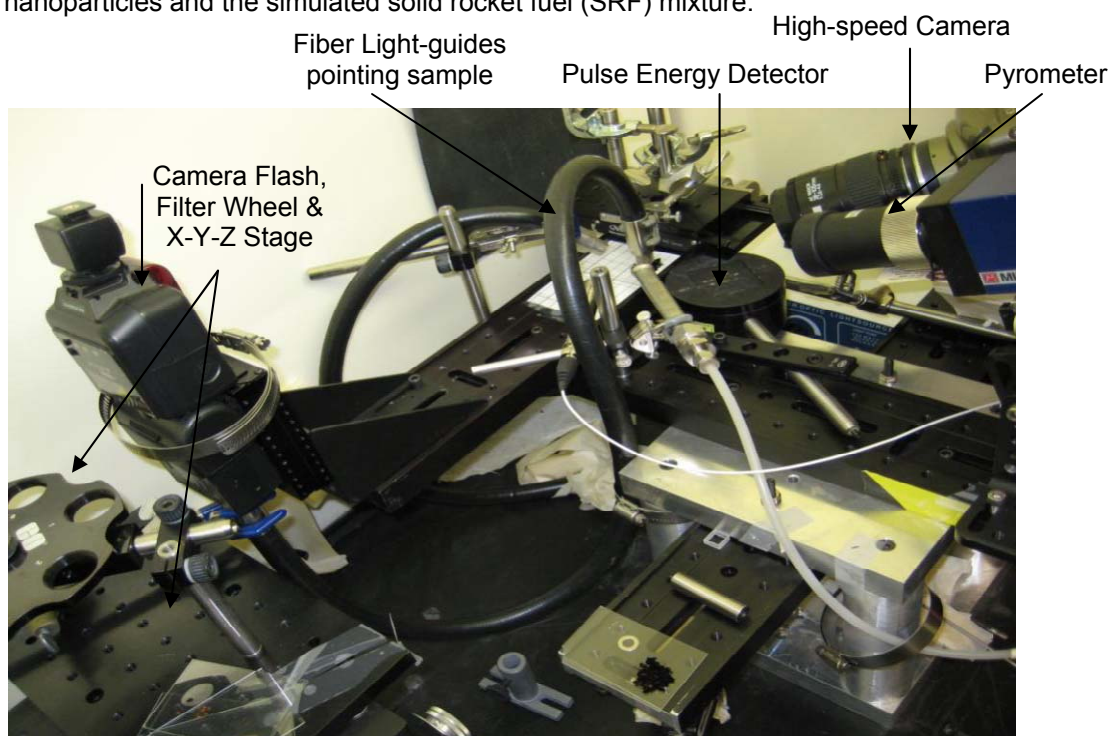
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variation of light intensity. Samples of as grown (“raw”) SWCNTs with different nanoparticle Fe content were purchased from Unidym Corp, Houston, Texas. A complete experimental setup is shown in Figure 1.

The data acquisition system Win600, a 16 channel digital scope/DAQ system from Hi-Techniques, was equipped with a photodiode detector as a trigger. The light from the camera flash provided a synchronization pulse through a digital delay/pulse generator. The DAQ system allows the synchronization between all of the detection systems in addition to registration of the signal for the photometer and any additional hardware such as the pyrometer. The entire experimental setup was housed inside a fume hood for safe handling of the CNT samples.

A high-speed pyrometer with a 6  $\mu$ s response time, model number KGA 740 HS, from Mikron Infrared Inc. of Oakland, NJ, covering from 300-2300° C, was used to determine the temperature of a millimeter size spot of the sample that was a few millimeter in size, which provided an instantaneous temperature measurement of the sample during the process leading to photoignition of CNTs as well as measurement of the onset of the ignition, the ignition temperature, and the burn duration of any other solids in ignition capsule such as the aluminum nanoparticles and the simulated solid rocket fuel (SRF) mixture.



**Figure 1**– The experimental setup for the study of photoignition effect in SWCNT. Digital camera and the fiber coupling for the spectrometer are not shown. A sample on a 2"x3" glass slide is also shown in the lower part of this picture.

A much simpler setup that is referred to as the fuel ignition apparatus was used to ignite a liquid spray or a simulated solid fuel via photoignition of CNT. The liquid spray was produced with a Vortex ultrasonic atomizing nozzle (UAN) from Sono-Tek Corp. A flow of gaseous oxygen was used to assist the photoignition. This setup utilized the aforementioned Canon 580 EXII light source, or a commercially available disposable camera flash, from Fujifilm or Kodak. The Canon 580 flash provided a fluence as high as 5J/cm<sup>2</sup> per flash at its highest setting (~7 ms duration), while the disposable camera flash provided 0.5-0.7J/cm<sup>2</sup> per flash with a typical duration close to 1 ms.

The above setup was used for photoignition of encapsulated CNTs, with nanoparticle Fe content between 43-51 Wt%. CNT samples were encapsulated with no additives as well as those

with added aluminum nanoparticles and/or SRF containing metal powders (such as aluminum) and solid oxidizers (such as ammonium perchlorate) cast in elastomeric polymer.

Phantom V7.1, a high-speed camera from Vision System, which is capable of capturing up to 4800 picture/s at full-frame, was used to capture the images of the photoignition process from the start of the flash of light and the sequence of the dynamics of the ignition process for characterization purposes.

In the photoignition process it is desirable to ignite the CNT samples with as little light fluence as possible, therefore minimum ignition energy (MIE) was used as a figure of merit for the CNTs ignition, as the main photoignition agent. In order to measure MIE two identical Canon camera flash sources were utilized through a Y-shape randomized fiber bundle. The electronics were in place to make sure that the two light sources are in sink so that the sample would receive a single light pulse with about two times more fluence than a single source. The flash sources were set at one of the 22 available power settings of a Canon camera flash (M1/1, M1/2, M1/4 to M1/128 and the steps in between), which also dictated the pulse duration of the flash between 0.1 to 7 ms. The light fluence was controlled through the Z-stage setting of the XYZ stage, starting from a value much lower than was needed for photoignition.

MIE was determined as the fluence was progressively increased until the onset of ignition was visually observed. The onset of ignition was easily identifiable for most samples. However, still photography was used for the observation of the appearance of ferrous oxide as the sign of the onset of photoignition. In order to make sure that the repeated exposure did not affect the onset of ignition, MIE for each sample was verified through repeated tests with an exposure slightly below the value of MIE that was found in the preliminary measurements.

The measurement of the fluence for MIE was provided by the pulse energy meter through a linear translation stage so that the exit of the fiber bundle could be easily moved from the sample area to the sensing area of the meter for the measurement of the energy/pulse at each step of the experiment. This practice ensured that the measured pulse energy was the same as what was seen by the sample.

In the liquid fuel ignition experiments the encapsulated CNTs (ignition capsule) were held close to the path of the spray from the ultrasonic atomizer so that upon ignition and rupture of the capsule the fuel and the burning particles impinge upon one another. Figure 3 shows a picture of the fuel spray and gaseous oxygen impinging flow. The camera flash was brought very close to the ignition capsule so that it would receive as much of the flash energy as possible. The photoignition and subsequent fuel ignition process was captured by a high-speed camera, at a rate of 500-2000 frames/second (FPS).

In the SRF ignition tests the ignition capsule was held near the top of a glass container, typically a beaker or an open-mouth jar of a volume close to that of a test solid rocket motor. The camera flash was brought close to the ignition capsule and induced photoignition. The ignition of CNTs causes the capsule to burst open and inject burning particles toward the SRF shavings that were placed within the container and cause them to ignite, while the process was captured by the high-speed camera through the glass. Subsequently, a ball of fire was produced that filled the entire container within a few milliseconds and continued burning until the SRF was completely consumed.

## **Sample Preparation**

### ***Encapsulated CNT***

It was observed that partially wet samples required substantially higher light fluence to produce photoignition. This made photoignition of the fuel spray very difficult, because the CNT sample needed to be close to a fuel spray in order to produce fuel ignition by the photoignition of CNTs. Two methods were devised to keep the CNT sample dry in order to overcome this

problem. The first method was the encapsulation of CNTs and the second one was lamination of CNTs along with a solid oxidizer(s) in a transparent polymer. Both methods proved to be effective, but the encapsulation was more controllable and repeatable and it was used for the ignition of the fuel spray.

Encapsulated CNT samples were prepared by placing the samples in a transparent plastic container 0.75 cm<sup>3</sup> in volume (D=7 mm L=20 mm). If there are enough CNTs in the container the gaseous byproducts of the photoignition of CNTs (mostly CO<sub>2</sub> or CO) would pressurize the capsule beyond 30 PSI and it would rupture and release its burning contents at the vicinity of the fuel spray and cause ignition of the spray.

## Results and Discussion

### *Ignition of SWCNTs*

In an earlier work<sup>13</sup> we investigated ignition characteristics of as grown SWCNTs as a function of flash pulse duration, wavelength of light, and Fe content. We also studied ignition characteristics of compacted SWCNTs samples in air and in an oxygen-rich environment. These experimental results indicate that the purified samples were relatively less likely to produce photoignition i.e. they required higher minimum ignition energy (MIE). It is not clear whether this was due to less Fe content in the sample or to reduced impurities in the samples. Electron microscopy images<sup>14</sup> of the raw and purified CNT samples suggest that substantial nano-scale impurities may be present in these samples as expected.

Table 2 shows the MIE for samples grown under nearly identical conditions by the same vendor that have different Fe contents. Samples R0554 and R0215 were used in the experiments that are subsequently reported in this paper, unless stated otherwise.

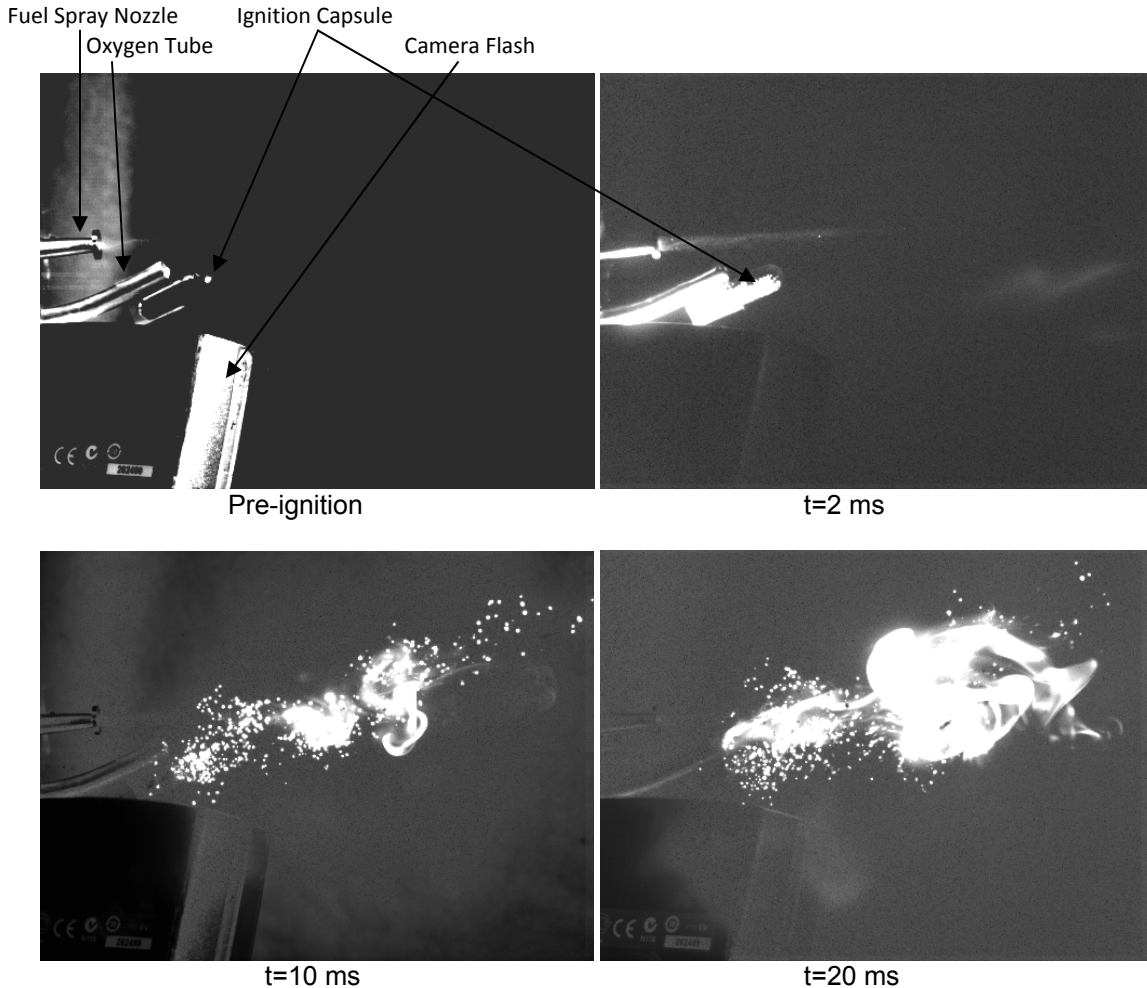
**Table 1.** Minimum Ignition energy for SWCNT samples with different Fe content in air

Sample Code	Fe Content of Sample (Wt%)	Sample Appearance	Min Ignition Energy Fluence (mJ/ cm <sup>2</sup> )
R0554	50%	like soot, velvet black, fluffy	69±7
R0215	43	like soot, velvet black, fluffy	71±7
R0532	39	like soot, velvet black, fluffy	84 ±10
R0220	30	like soot, velvet black, fluffy	100 ±10
R0471	18	like soot, velvet black, fluffy	180±13
P0232	12	gray/black powder	>750*
D0381	5	gray/black powder	>750*

\* no ignition was observed up to the maximum available fluence for the pulse duration setting

### *Ignition of a Liquid Fuel Spray*

Encapsulated CNT were used with the fuel ignition setup in testing the photoignition of the liquid fuel in order to keep the CNT samples dry. If there is enough CNT in the container the photoignition of CNTs would pressurize the capsule and make it burst and release its burning contents at the vicinity of the fuel spray, which resulted in the ignition of the spray with a reasonable rate of success, typically better than 50%. However, mixing the fuel and the oxidizer was more effective for some fuel than the others. Some liquid fuels such as methanol have a relatively wider range of ignitable mixture ratio as compared to typical hydrocarbon-based liquid rocket fuels such as RP1, thus it was easier to ignite them by photoignition of CNTs.



**Figure 3-** Photoignition of Hexane+Acetone (50% each) in a sequence of images taken at 2000 frames per second (FPS), where  $t=0$  indicates the peak of the flash. In this case gaseous  $O_2$  was introduced in an impinging flow at a rate of 7 Lit/min. The liquid fuel was introduced at a rate of about  $2 \text{ cm}^3/\text{min}$  through a programmable syringe pump. These images were captured at 2000 FPS and exposure time of  $490 \mu\text{s}$ .

Fig. 3 shows the photoignition process for an impinging liquid fuel liquid spray and  $O_2$  as induced by a camera flash. This figure shows the following sequence of events:

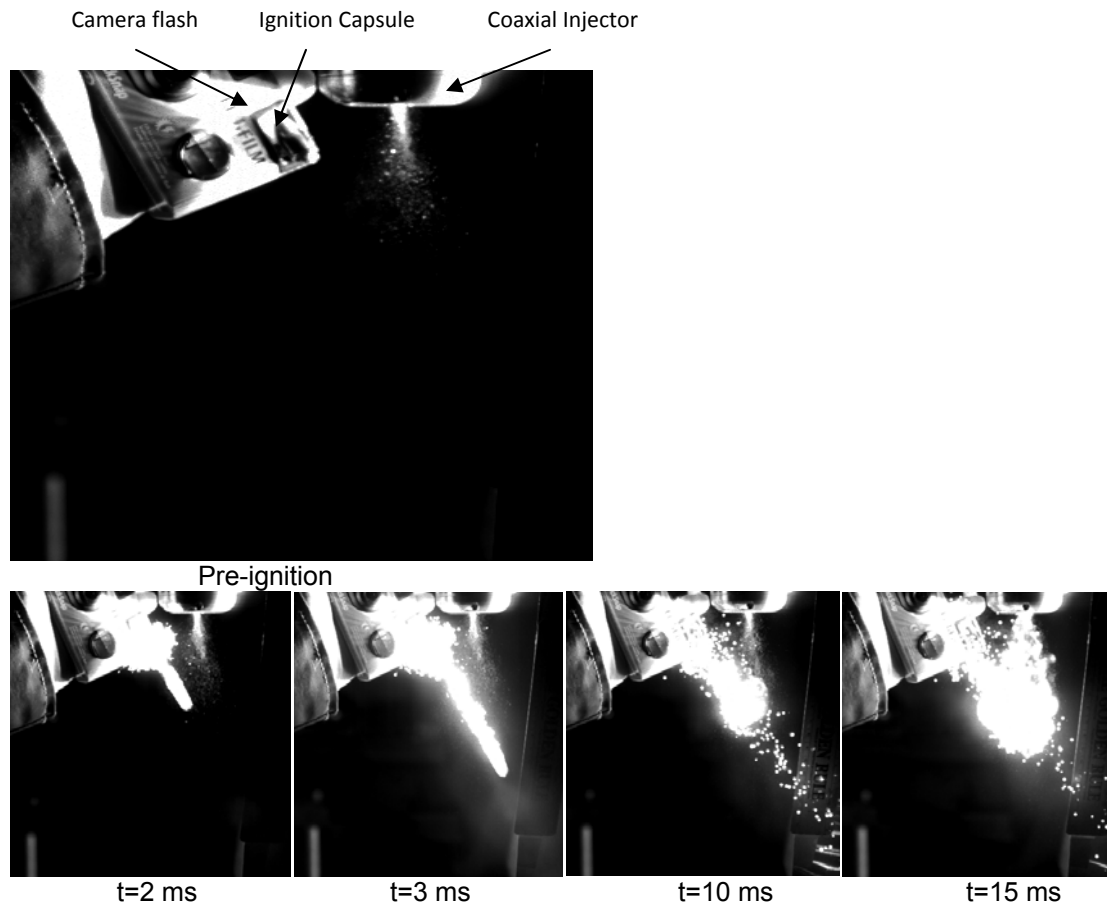
- A capsule ( $D=7 \text{ mm}$ ,  $L=20 \text{ mm}$ ) containing SWCNTs was attached to the oxygen tube located near the exit of the ultrasonic atomizer
- The camera flash was held close to the oxygen tube and the encapsulated CNTs
- The flash was activated and lasted for 1 ms. Within about 2 ms from the peak of the flash, the CNT capsule ruptured due to the high pressure of the gaseous byproducts (mostly  $CO_2$  &  $CO$ ) inside it.
- At  $t= 5 \text{ ms}$  burning particles intersected the spray mixture at a speed of about 20 m/s.
- At  $t= 10 \text{ ms}$  multiple ignited spots appeared across the volume of the fuel/oxidizer mixture, as it is evident from the image a small scale volumetric fuel ignition has occurred.
- At  $t= 20 \text{ ms}$  the entire spray was in flame.

For the liquid fuels with a relatively wide range of ignitable mixture ratios the approach that is shown in Fig. 3 resulted in a relatively high rate of success (better than 50%) in ignition of

the spray by CNT. However such an outcome could not be guaranteed for other fuels such as kerosene due to an unfavorable fuel and oxidizer mixture ratio.

In order to provide an effective mixing of the fuel spray and gaseous oxygen, a modified gas delivery injector was designed and built for the ultrasonic atomizer to provide a concentric gas flow. Computational fluid dynamics (CFD) was employed to create a model for the details of the design. The CFD work will be presented in a separate publication. The new design included a gas chamber and a number of specially shaped fins in order to produce substantial swirl at the coaxial gas outlet of the ultrasonic atomizer.

As a result, a turbulent flow was observed near the annular gas exit of the ultrasonic injector/atomizer, which produced effective mixing of the oxygen and the fuel micro-droplets. Fig. 4 shows a typical fuel spray with gas mixing function that was provided by the designed swirl inducing stator and the gas delivery injector.



**Figure 4-** Photoignition of Hexane+Acetone (50% each) in a sequence of images taken at 2000 PPS where  $t=0$  indicates the peak of the flash. In this case gaseous  $O_2$  was introduced in a coaxial flow at a rate of 7 Lit/min with a swirl motion in order to produce an effective fuel and oxidizer mixing. The liquid fuel was injected at a rate of about  $3 \text{ cm}^3/\text{min}$  through a programmable syringe pump. These images were captured at 2000 FPS and exposure time of  $490 \text{ } \mu\text{s}$ .

Fig. 4, shows the result of fuel ignition with the improved fuel and oxidizer mixing approach. There is the following sequence of events:

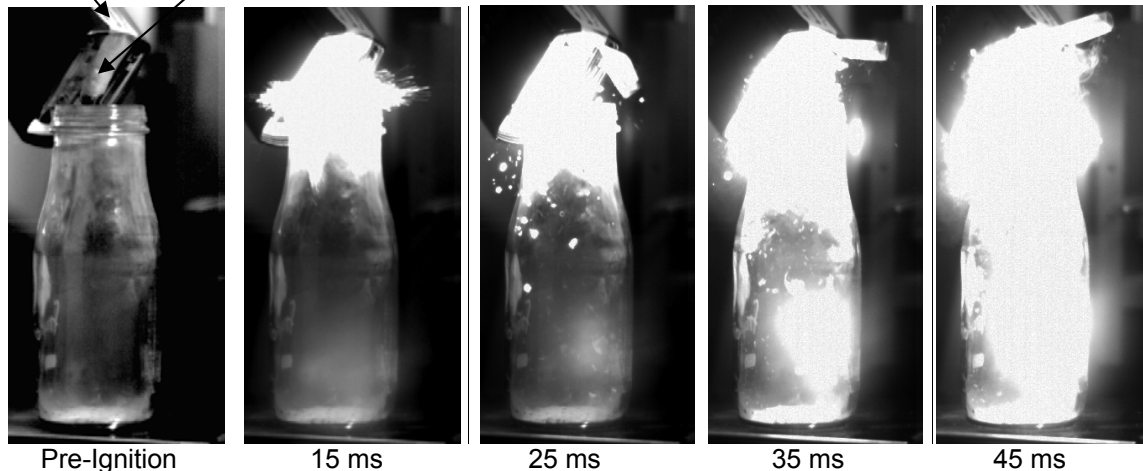
- A capsule (D=7 mm and L=20 mm) containing CNT and aluminum nanoparticles was attached to the flash of an instant camera and held close to the exit of the ultrasonic atomizer.
- The camera flash was activated and it lasted for approximately 1 ms. 2 ms after the peak of the flash the capsule ruptured along the seam and the burning CNT “particles” were injected in the path of the fluid spray and O<sub>2</sub> mixture.
- At t = 3 ms after the flash, burning particles were intersecting the spray mixture.
- At t = 10 ms there was a volume of ignited fuel mixture
- At t = 15 ms the entire spray was in flame. In this case, the top of the flame at the exit of the injector has a diameter of about 1 cm.

The approach in Fig. 4 greatly improved the chance of a successful fuel ignition by CNTs (better than 90%) due to improved mixing of the fuel and the oxidizer. The choice of liquid fuel mixture was to get a better combustible due to acetone and produce a highly visible flame for imaging from Hexane. In this case, the speed of photoignition has improved as well. We believe with a proper fuel and oxidizer mixing the fuel ignition by can be further improved toward 100% and this approach can lead to a distributed ignition similar to that of Fig. 3.

### ***Fuel Ignition for Simulated Solid Rocket Fuel***

Electrical ignition is widely used in solid rocket motors. Typically, the electrical hot wire ignites an explosive bag which rapidly produces hot gases to achieve the required pressure and temperature for the ignition of the solid rocket fuel. Typically the solid fuel is a special blend of the fuel and the oxidizer that is cast into a rubber like hollow cylinder and is designed to burn from inside out under high pressures and temperatures.

Camera Flash Ignition Capsule



**Figure 6-** Photoignition of CNT/MRF/SRF with a specific concentration exhibits a relatively large ignition plum which fills the glass container that is similar to a test rocket motor in size. These images were captured at 1000 FPS.

We have successfully tested photoignition of simulated solid rocket fuel (SRF) through photoignition of CNT. SRF was specially formulated to produce a rapid combustion similar to a typical rocket fuel, while being non-explosive due to safety considerations. Commercially available model rocket fuel (MRF) used for hobby rocketry was also used for photoignition with similar results with and without encapsulation. Fig. 6 shows an example of the photoignition of the SRF that was encapsulated with CNTs that is attached to the camera flash at the top of a glass container. In this cases, addition of a fraction of a gram of millimeter size SRF to the



encapsulated mix increased the size of the ignition plume, as well as its burn temperature and its duration.

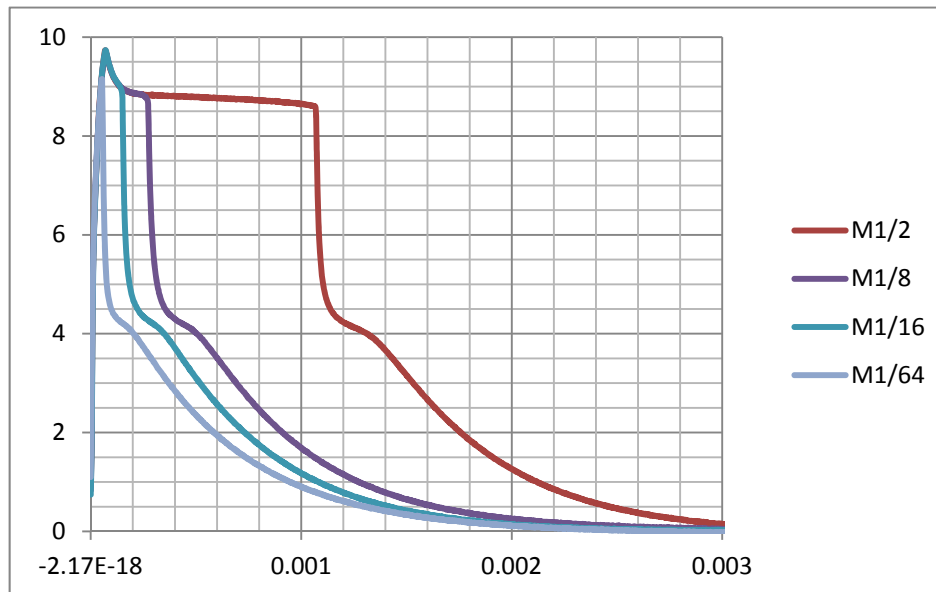
### Tailoring Ignition Parameters for Rocket Ignition Applications

In certain cases, the addition of a few milligrams of aluminum nanoparticles (Al-NPs) to the encapsulated CNT improved the probability of a successful ignition of the SRF. The Al-NPs had a nominal size of 18 nm and were passivated in order to prevent it from oxidizing. During the process of testing a mix of CNT, Al-NPs and grains of SRF, it was noted that different proportion of three ingredients produced an igniter with different ignition properties such as ignition temperature, duration, pressure ramp up and ignition delay. A typical mixture includes a few milligram of CNT, a small fraction of a gram of Al-NPs and a fraction of a gram of SRF in a  $0.75 \text{ cm}^3$  transparent capsule.

The function of CNT is to provide photoignition at a low light level (less than  $0.1 \text{ J/cm}^2$ ), while it produced substantial amount of gas to pressurize the capsule. The two part capsule that was used in this study typically ruptured at about 30 psi. Al-NPs was easily ignited by CNTs and pyrometer data indicate that it produced a high burning temperature (exceeding  $1200^\circ \text{C}$ ), which lasted well beyond 200-ms. Although, Al-NPs showed photoignition at high photon flux ( $> 0.6 \text{ J/cm}^2$ ) without CNTs, they produced very little gaseous byproducts, not enough to contribute to the pressure build up within the capsule.

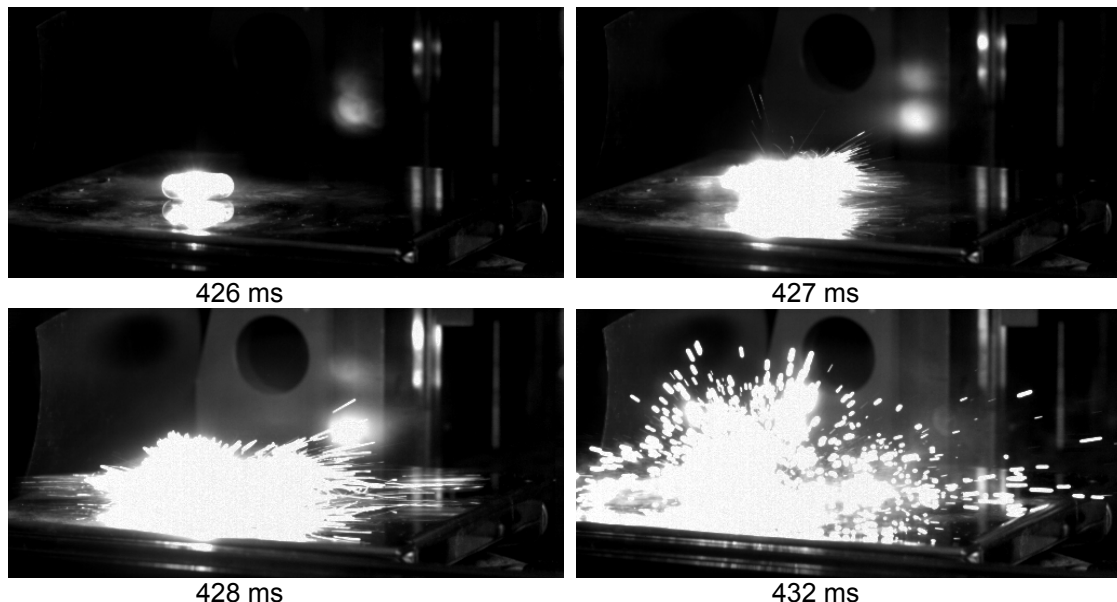
Al-NPs burn much longer than CNTs and at high enough temperature to ensure the ignition of the SRF particles. Pyrometer data indicates that SRF particles burn at a very high temperature (approaching  $2000^\circ \text{C}$ ), they produce a substantial amount of gas and burn for substantially long time, well beyond 500-ms, depending on their size and their surface to volume ratio.

Fig. 7 shows a pyrometer response of photoignition of an ignition capsule for four different samples. The voltage reading, the vertical axes, indicates the temperature as a function of time and it shows that the burn duration changes with the ingredients within the ignition capsule.



**Figure 7.** Shows the response of the pyrometer during the ignition of four different encapsulated samples each with a different solid mixture. The voltage reading on the vertical axes is a measure of burn temperature as a function of time.

It was verified through many tests that a combination of the three ingredients (CNTs, Al-NPs, SRF) provides the capability of tailoring the burn temperature, pressure ramp up and burn duration for different applications. Fig. 8 shows an example of the photoignition of an ignition capsule containing three solid ingredients with an unusually long delay in the combustion. The first image was taken 426 milliseconds after the peak of the camera flash.



**Figure 8-** shows photoignition of CNT/Al-NPs/SRF with a relative concentration so that it exhibits a substantially long delay in the final ignition and rupture of the ignition capsule (D=7 mm, L=20 mm). The first image shows the ignition capsule 426 ms after the peak of the camera flash just before it ruptures. These images were captured at 2000 FPS and exposure time of 490  $\mu$ s.

## Conclusion

Photoignition of different fuel sprays was achieved through photoignition of encapsulated CNT, which injects burning particles into the path of the spray. A similar approach was used to ignite the SRF through encapsulation of aluminum nanoparticles along with CNT. An ignition system based on photoignition of CNTs offers the following advantages:

- It is light weight and compact, a typical ignition system has a weight of less than 100-gr and it occupies less than 50-cm<sup>3</sup>.
- It is low cost i.e. typical ignition system is so inexpensive that it provides affordable redundancy with nearly zero effect on the total cost of the rocket.
- It is safe and reliable, the implemented ignition system is based on battery operated compact Xe-lamp used in pocket camera and it has three layers of electrical safety.
- It is easy to implement, the compact system can be used instead of, or along with, any other ignition system such as spark plugs and electrical match (as a back-up).
- It is scalable, while the application of the photoignition has been demonstrated for small scale fuel spray and simulated solid propellants in this report, it can be easily scaled up to be used for large rockets.
- Space applications, though the demonstrated fuel ignition experiment was achieved through the use of CNTs and gaseous/environmental oxygen, the preliminary tests have shown that the ignition system can be implemented with solid oxidizers encapsulated in impermeable layers for space applications.

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13. To be published separately.
14. See for example <http://www.nano-c.com/nanoprod.html>